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Investigation of the Compressive Material Properties of PZT and PMN

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PREFACE

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13. ABSTRACT (Maximum 200 words)

In U.S. Navy sonar transducers, the electroactive ceramics are subject to mechanical compressive stresses. However, there has been scant research to examine the mechanical effects of compressive loads on electroactive ceramics. The research reported herein measured material properties (compressive strength, Young's modulus, and Poisson's ratio) and studied the behavior in compression and under short-circuit conditions of five commercially available lead zirconate titanate (PZT) and lead magnesium niobate (PMN) compositions. The electrostrictive PMNs are the weakest, and the piezoelectric PMN is the strongest, with the PZTs falling in between. The strength differences are attributed to differences in the fracture toughness values. The PZT ceramics exhibit nonlinear stress-strain curves. Considerable hysteresis and the presence of a remnant strain exist upon unloading. This behavior is associated with the movement of 90° domains.

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INVESTIGATION OF THE COMPRESSIVE MATERIAL PROPERTIES OF PZT AND PMN

INTRODUCTION

The U.S. Navy has had good results for years using lead zirconate titanate (PZT) ceramics as the active element in sonar transducers. However, as the requirements on sonar systems change due to a shift from blue water to littoral areas, performance gains need to be realized in the electroactive ceramics. Performance gains in PZT can be realized by increasing the magnitude of the electrical field used to drive the transducers. Larger electric fields will necessitate the application of larger compressive stresses on the stack of PZT ceramic elements during transducer fabrication. The mechanical load that is applied during fabrication and maintained throughout the lifetime of the transducer is sufficiently large (in magnitude) to avoid the development of tensile stresses during sonification. To support the use of PZT under these more severe mechanical conditions and to aid the transition of developmental materials such as lead magnesium niobate (PMN) into fleet transducers, the mechanical properties of these materials need to be elucidated.

Within the past several years, the experiences of U.S. Navy researchers have highlighted the importance of understanding the mechanical behavior of electroactive ceramics. Numerous failures of transducer elements and test specimens fabricated from electrostrictive PMN occurred in a variety of situations, from stack glue-up to supposedly nondestructive electromechanical characterization involving mechanical loading. Based upon experience with PZT, these failures were unexpected and suggested problems with the mechanical strength of PMN.

A study (reference 1) comparing the flexural strength of several PMNs to several PZTs reported that the flexural strength of that generation of PMN was indeed very low. The PZTs were 37% to 157% stronger than the PMNs, depending upon the specific materials being compared. Fractographic investigations revealed large flaws in the PMNs. Fracture toughness analysis predicted that if the flaw size was reduced an order of magnitude (a realistic goal from the perspective of both processing and microstructure), the strengths of the PMNs would be comparable to the PZTs. Armed with this information, one manufacturer was able to raise the strengths of its PMNs by 44% in 3 months.

Research on PZT and PMN ceramics must also examine the effect of compressive stress on mechanical performance. The application of compressive mechanical stress has received some attention (references 2 through 5), but the focus has been the effect on dielectric and electromechanical properties. Even when the focus shifts more heavily toward mechanical properties (reference 6), not all the relevant testing issues are addressed.

The structural ceramics community has laid an extensive groundwork for the accurate mechanical testing of brittle materials. Much research has been dedicated to the development of test methodologies for the study of compressive properties (references 7 through 9). Proper design of the test specimen and of the loading fixture is paramount for ensuring both the desired stress state in the sample and the repeatability and accuracy of the test technique.

The work reported herein utilized a compression test technique that was developed by the structural ceramics community and is the foundation of a proposed American Society for Testing and Materials (ASTM) standard to study the behavior of electroactive ceramics under a uniform compressive stress state and known electric field conditions. The research focuses on commercially available PZTs and PMNs that are in use or under investigation for future use by the U.S. Navy. The compressive strength, Young's modulus, and Poisson's ratio at several load rates were measured on specimens having a reduced gauge section geometry optimized to minimize variations in the stress state through the gauge section.

EXPERIMENTAL PROCEDURE

The compressive properties of five electroactive ceramics were investigated: (1) an electrostrictive PMN containing 10% lead titanate (PT) and doped with 1% lanthanum (PMNw/La) from TRS Ceramics in State College, PA; (2) an electrostrictive PMN containing 10% PT doped with 3% barium (PMNw/Ba) from TRS Ceramics; (3) an unpoled piezoelectric PMN (unpoled PMN) designated EC-98 from EDO Corporation in Salt Lake City, UT; (4) an unpoled Navy Type III PZT designated EC-69 from EDO Corporation; and (5) a poled Navy Type III PZT designated EC-69 from EDO Corporation.

When performing compression tests, it is essential to guarantee that the desired stress state occurs in the test specimen. In compressively loaded specimens, deviations from a pure compressive stress state can be created by the boundary conditions on the loaded faces. Test specimens are typically loaded using materials that will not yield or break during testing. This situation necessitates that there is a mismatch of material properties at the specimen's loaded faces. According to St. Venant's principle, this property mismatch leads to a significant change in the internal stress state of the specimen. The distance over which this undesired stress state exists (measured perpendicularly into the specimen from a loaded face) varies, depending upon the boundary condition on the loaded face. However, an accepted rule of thumb states that the distance is usually one to three times the length of the loaded face.

To obtain a region of material having the desired stress state with a constant cross-section specimen, necessitates a large length-to-width ratio. Such specimens can be prone to buckling and to failure in the region with the undesired stress state. An alternate and commonly employed solution is to design specimens with a reduced gauge section. Tracy (reference 7) and Dunlay et al. (reference 8) optimized a large and a small dumbbell-shaped specimen, shown in figures 1a and 1b, for compression testing of structural ceramics. The current research verified through finite element analysis that the variation in the stresses through the gauge section of these specimens is less than 7%. This is an excellent result. Thus, these two dumbbell-shaped specimens were employed for the mechanical testing of electroactive ceramics.

Deviations from a pure compressive stress state can also be caused by misalignment of the applied load. To minimize such misalignments, a double ball-joint fixture was used to load the components of the load train and the specimen. The load train consisted of one insert and one load block on either side of the specimen, as shown in figure 2. To further control misalignment, specimens were machined with the tight tolerances given in figure 1. These tolerances were also used in machining the inserts and load blocks.

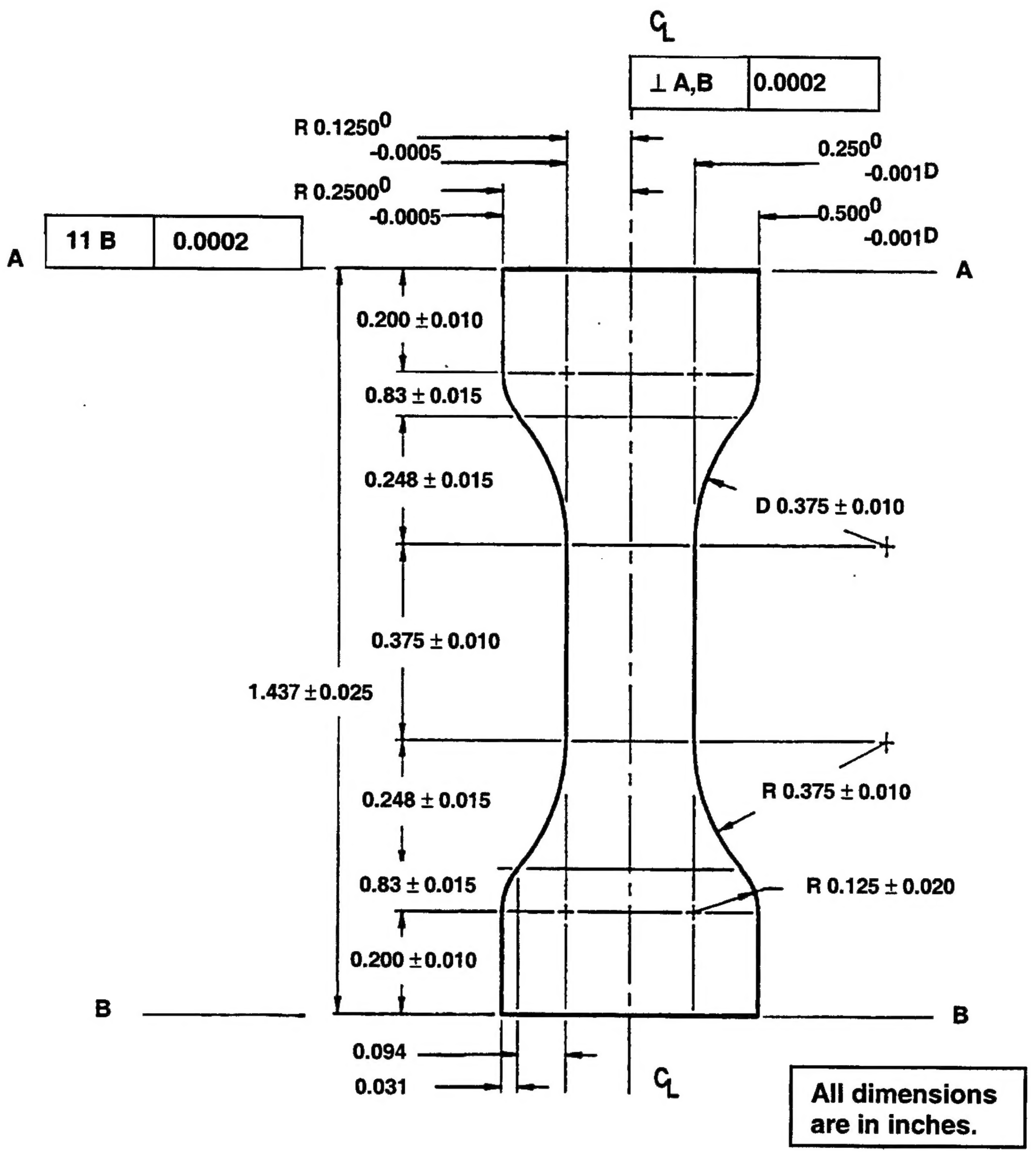

Figure 1a. Large Dumbbell-Shaped Compression Specimen

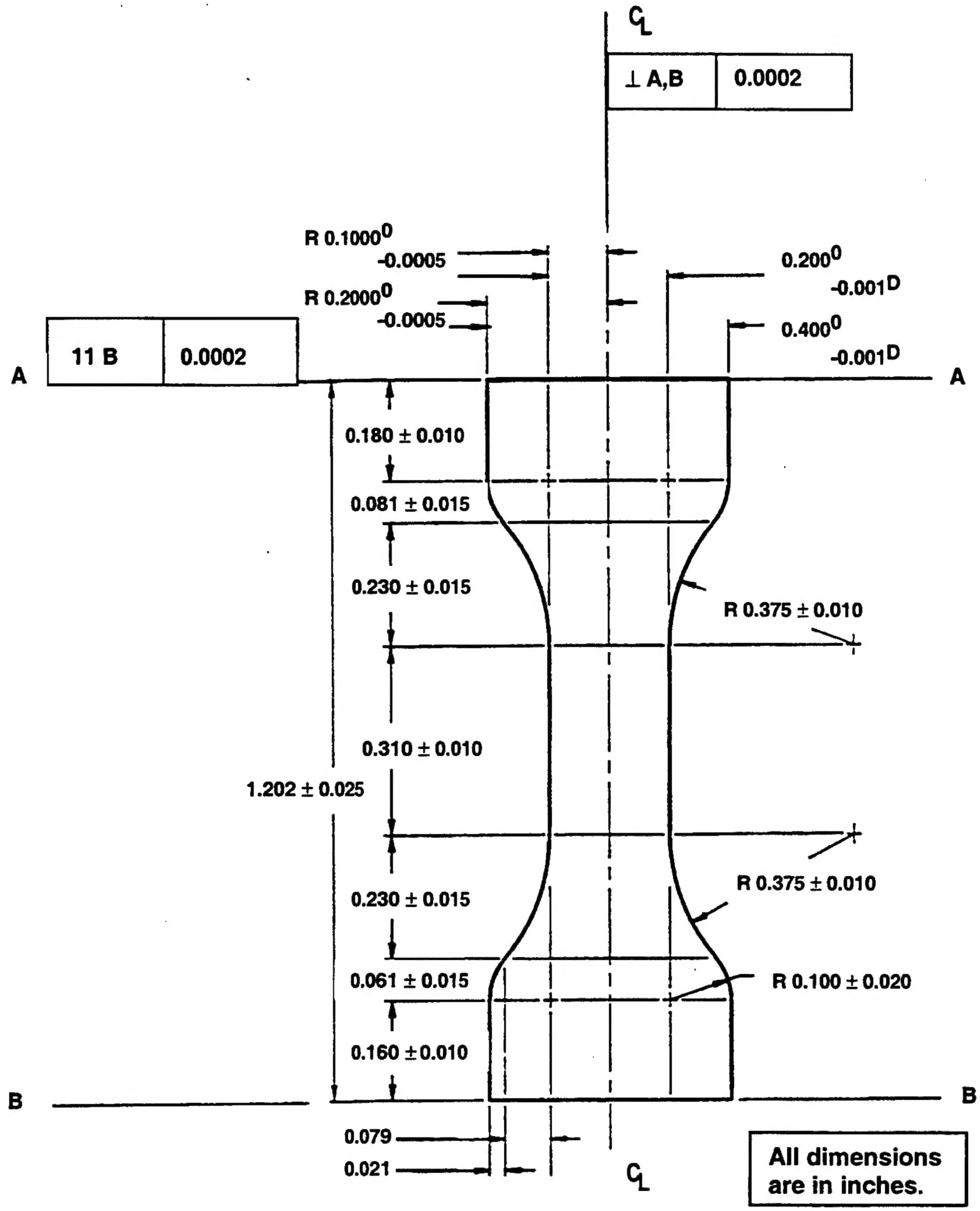

Figure 1b. Small Dumbbell-Shaped Compression Specimen

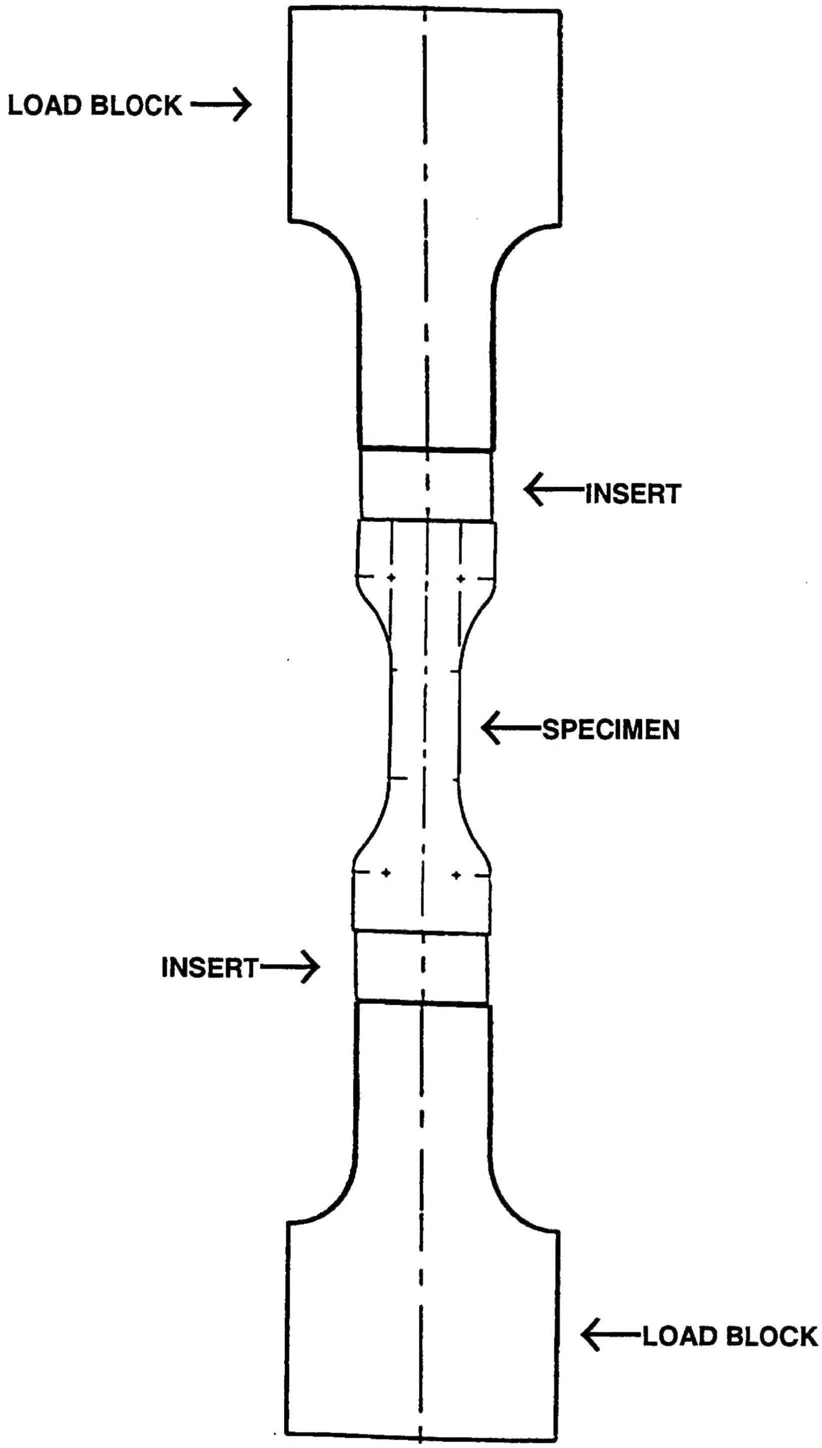

Figure 2. Load Train and Specimen

The development of tensile stresses at or near the loaded faces, which would lead to premature specimen failure, is avoided by the proper selection of insert materials. According to Tracy (reference 7) and Dunlay et al. (reference 8), slight radial and circumferential compressive stresses at the loading faces are created when the insert material has a Young's modulus and Poisson's ratio that satisfy the equation (P/E) specimen/(P/E)insert ≥ 1, where P is Poisson's ratio and E is the lateral Young's modulus of the specimen and insert materials. In the current research, finite element analysis of various material combinations indicates that this equation has an upper bound of about 2 for the present configuration. Using both this equation and finite element analysis as a guide, high-strength beta-c titanium from G&S Titanium was chosen as the insert material. The load blocks were machined from alumina designated AD94 available from Coors Ceramic Corporation in Golden, CO.

The same vendor machined all the test specimens and load train components. The two electrostrictive PMNs were machined into large dumbbells, and the other three ceramics were machined into small dumbbells. In the case of the poled PZT, the specimens were machined with the poling direction perpendicular to the long axis of the specimen. Thus, during testing, the poling direction was perpendicular to the axis of compression.

On all specimens the end faces, where the compressive loads were applied, had electrodes. The application of three biaxial strain gauges, spaced 120° apart in the same cross-sectional plane in the middle of the reduced gauge section, allowed the measurement of both the Young's modulus and the Poisson's ratio. The gauged specimens were encapsulated to help retain the broken pieces of the test specimens for further analysis. The encapsulation procedure entailed priming the specimens with Dow Corning Z 6020 silane and then potting them with Composite Polymer Design 9130 polyurethane.

Each specimen and the other four load train components were positioned for testing using the procedure described in Tracy (reference 7) and Dunlay et al. (reference 8). Briefly, a machinist v block was used to align the five pieces on top of one another. Once in place, copper tape was attached to the two inserts, and the resistance was measured to confirm that a short circuit existed.

The specimens were tested in a servohydraulic test machine (Instron load frame model 1332 and control system 8500). The standard testing rate was 6 kN/min. To examine the effect of load rate on the mechanical properties, specimens were also tested at 0.6 kN/min and 60 kN/min. However, the presented results represent specimens tested at 6 kN/min unless otherwise specified. At least three specimens per load rate were tested to failure. Interrupted tests were also conducted. In interrupted tests, specimens were unloaded before failure to examine mechanical hysteresis in the materials.

RESULTS

The five electroactive ceramics exhibited a large range of strengths. The electrostrictive PMNs had the lowest compressive strengths of the five materials tested (see table 1). Despite the difference in dopant composition and quantity, the two electrostrictive PMNs had essentially the same strength (≈ 874 MPa or 127 ksi). In comparison, however, the process of poling had a significant effect on the strength of PZT. When the poling direction was oriented perpendicular to the applied compressive stress, the strength dropped 10% from 1045 MPa (152 ksi) for the unpoled ceramic to 944 MPa (137 ksi) for the poled PZT. The unpoled PMN, at a compressive strength of 1309 MPa (190 ksi), was 25% to 39% stronger than the PZTs, depending upon electrical history, and 48% stronger than the electrostrictive PMNs.

Table 1. Material Properties

Material	Compressive Strength at 6 kN/min (MPa)	Young's Modulus (GPa)	Poisson's Ratio
PMNw/Ba	877 ± 117	131 ±4	0.30 ± 0.02
PMNw/La	871 ± 43	132 ± 4	0.30 ± 0.02
PMN	1309 ± 41	111 ± 4	0.46 ± 0.02
PZT Unpoled	1045 ± 45	76 ± 1	0.43 ± 0.02
PZT Poled ⊥	944 ± 50	83 ± 4	0.44 ± 0.06

The stress-strain behavior of the materials varied greatly (see figure 3) as did their Young's modulus and Poisson's ratio (see table 1). Both of the electrostrictive PMNs responded linearly to the compressive loading up to failure. Their Young's modulus and Poisson's ratio values, 131 GPa (19 x 10^6 psi) and 0.30, respectively, were essentially the same. The unpoled PMN appeared to have two linear regions. The initial part of the stress-strain curve was linear to approximately 200 MPa (29 ksi). Beyond this stress, the stress-strain curve deviated to a slope higher than the Young's modulus of 111 GPa (16 x 10^6 psi). The unpoled PMN had the largest Poisson's ratio of 0.46.

The initial linear region of the PZTs was very small (see figure 3). The poled PZT was linear up to approximately 150 MPa (22 ksi), giving a Young's modulus of 83 GPa (12 ksi) and a Poisson's ratio of 0.44 (see table 1). The unpoled PZT was linear only up to approximately 60 MPa (9 ksi). The resulting Young's modulus was 76 GPa (11 x 10^6 psi), with a Poisson's ratio of 0.43.

The PZTs were the only two ceramics to exhibit hysteresis in the stress-strain curve when unloaded from large stress levels. For example, the poled PZT had a remnant strain of 0.0015 after being loaded from 65% of its failure strength (see figure 4). The unpoled PZT had a remnant strain of over 0.002 after being loaded from only 38% of its failure strength (see figure 5). The effect of load rate on the compressive strength of the ceramics was also investigated. For all five materials the compressive strength increased with increasing load rate. This is shown graphically in figure 6. The unpoled PMN showed the strongest response to a change in the load rate.

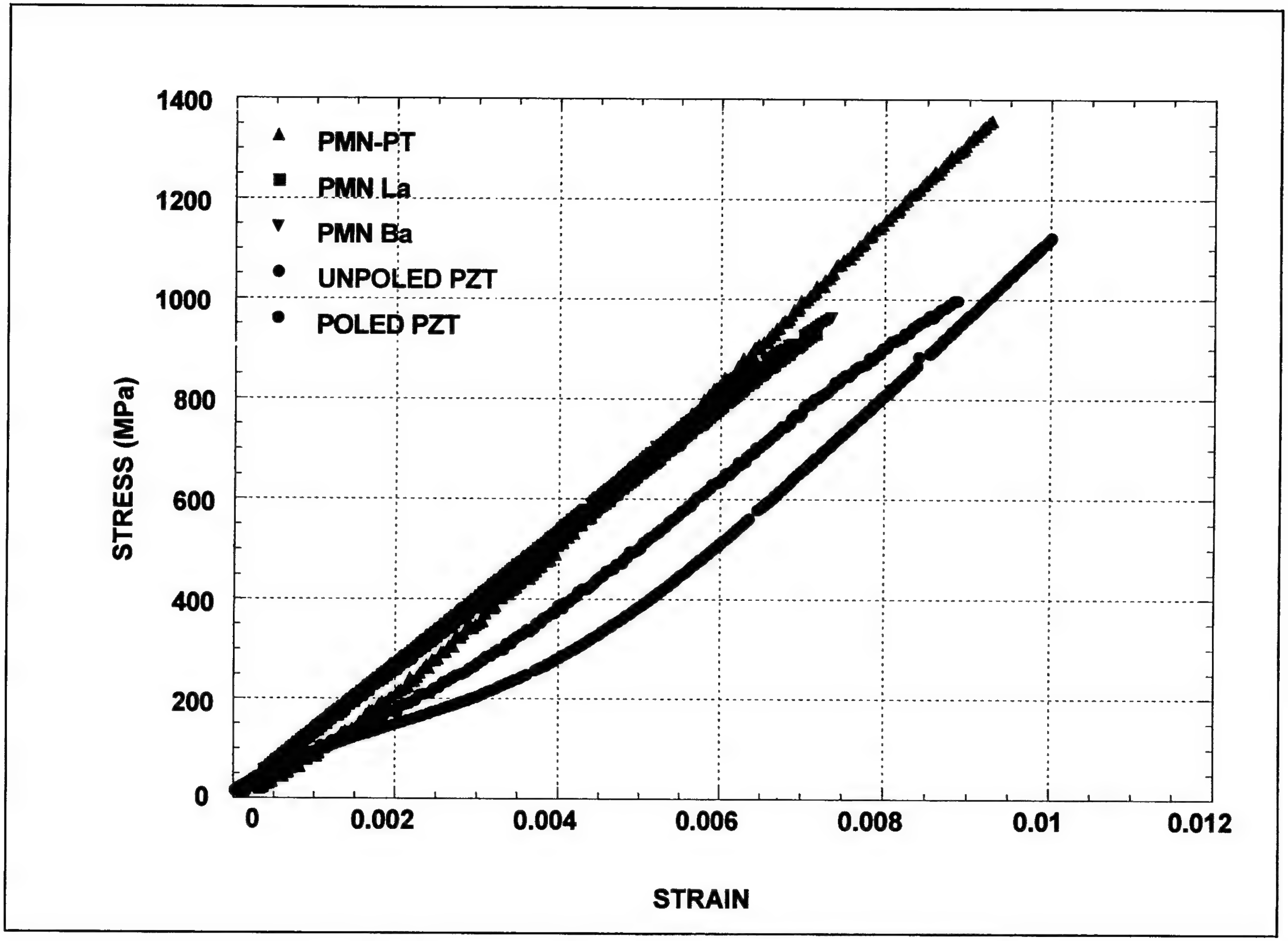

Figure 3. Comparison of Materials (Stress Strain Curves at 6 kN/min)

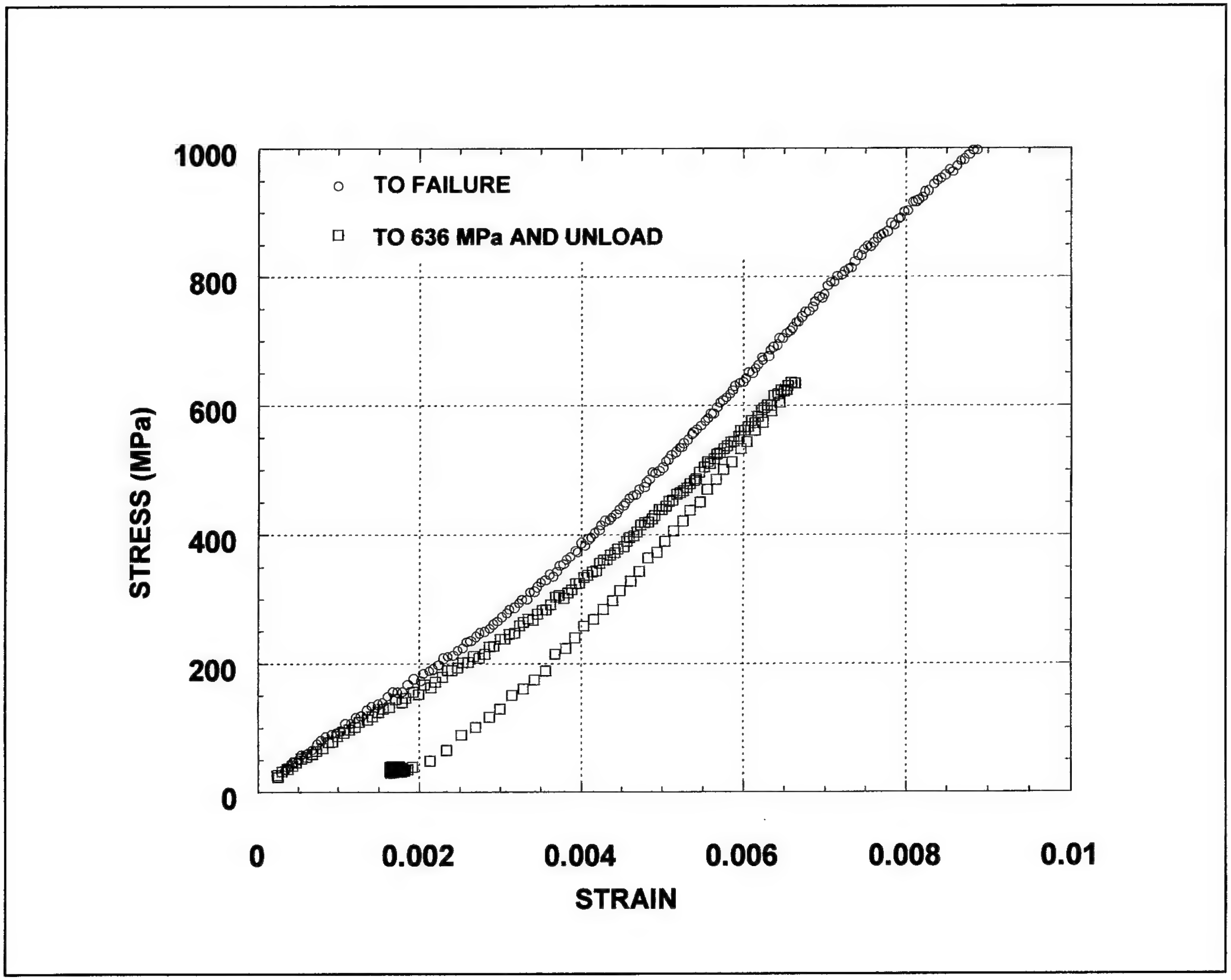

Figure 4. Poled PZT (Stress-Strain Curves at 6 kN/min)

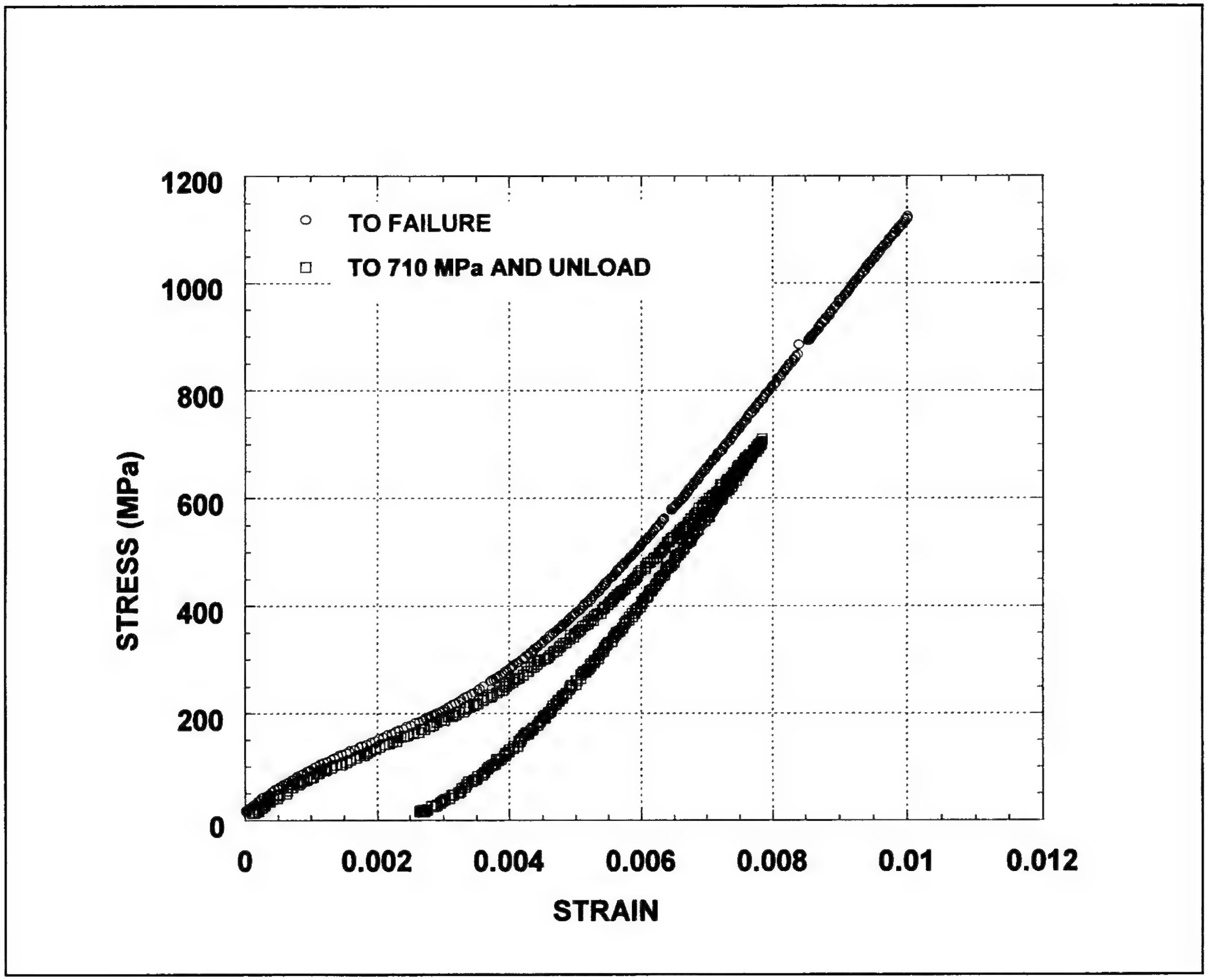

Figure 5. Unpoled PZT (Stress-Strain Curves at 6 kN/min)

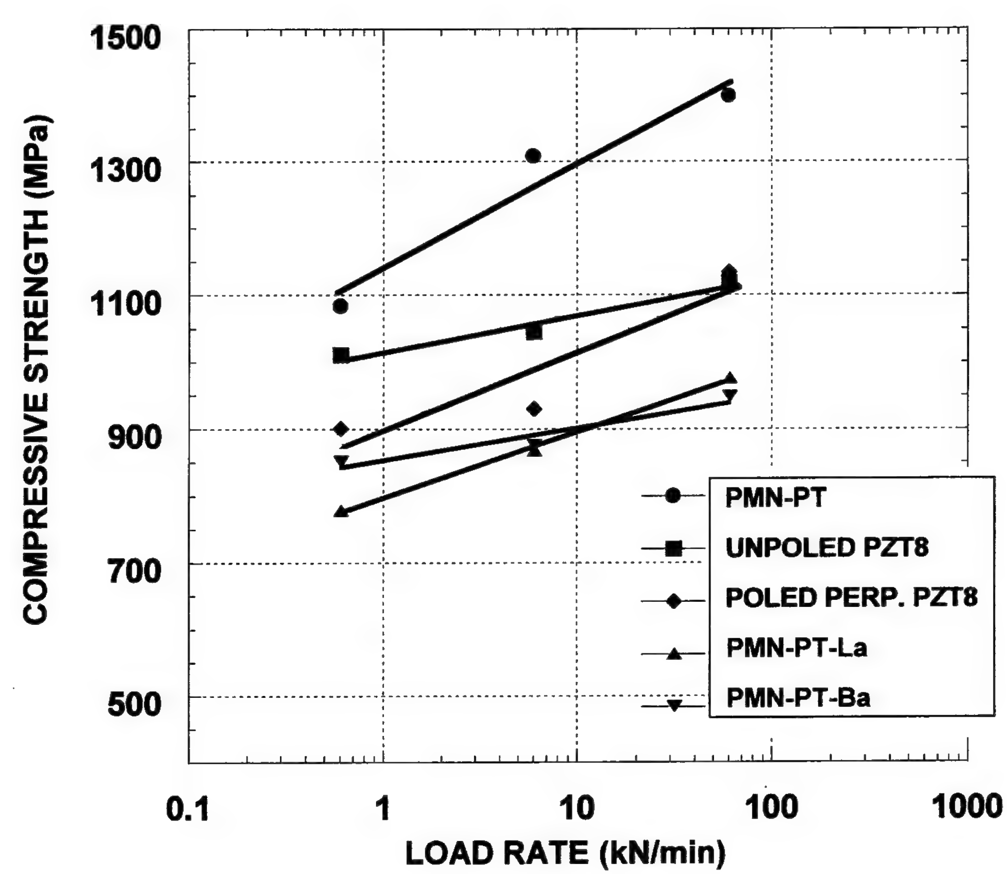

Figure 6. Compressive Strengths at Three Load Rates

DISCUSSION OF RESULTS

The two electrostrictive PMNs had the lowest compressive strengths of all five ceramics (see table 1). In flexural strength studies (references 1 and 10), comparing electrostrictive PMNw/Ba and PMNw/La to PZT (all materials from the same manufacturers as in this study), the PMNs also had the weakest strengths. Fractographic analysis identifying the native flaw at each fracture origin was able to guide processing changes that reduced flaw sizes and significantly raised the PMN strengths. Failure in compression led to pulverization of the test specimen. Thus, there was essentially no specimen left for post-mortem examinations to allow identification of the flaw that led to final fracture, its size, or its orientation.

Interestingly, the unpoled PMN proved to be, by far, the strongest of the five ceramics in compression. One possible reason for the high compressive strength is that the unpoled PMN had the largest fracture toughness value(s) (K_{IC} in tension and/or K_{IIC} in shear) of all five materials. Both K_{IC} and K_{IIC} were considered because a fracture criterion for failure in compression had not been established. Cracks do not grow in compression. Under far-field compressive loads, local tensile and shear stresses govern crack propagation. It is not known whether K_{IC} and/or K_{IIC} dominates the local fracture process. The local fracture process is stable because the bulk of the material is in compression. Failure occurs when K_{IC} and/or K_{IIC} for the material is exceeded locally *and* conditions for unstable propagation are achieved in the bulk.

A comparison of numerous studies in the literature reveals that various compositions of PMN have a lower K_{IC} than PZT regardless of its electrical state(references 10 through 14). Since studies of PMN have focused on electrostrictive compositions, a definitive statement on the K_{IC} of the unpoled PMN relative to PZT cannot be made. Measurements of K_{IIC} for these materials have not been made. However, the same vendor manufactured the unpoled PMN and the two PZTs. Thus, it is reasonable to assume that the native flaw population introduced during processing is similar, especially in size, for the unpoled PMN and the PZTs. This would imply that the unpoled PMN does indeed have the largest fracture toughness value(s). This possibility is further supported by the idea, suggested in the literature, that the fracture process in compression is not dominated by the native flaw population but rather by the initiation and coalescence of microcracks. The process of microcrack coalescence and final fracture would then be governed by the fracture toughness value(s) of the material.

A comparison of the PZTs reveals that the unpoled material is stronger. Since these PZTs are from the same batch of material, processing variations cannot explain the difference. The strength difference between the unpoled and poled PZTs can be explained based upon the effect of electrical history on the fracture toughness of PZT. Studies have shown that poling affects the K_{IC} of PZT as does the relationship of poling direction to the direction of crack propagation (references 10 through 14). The literature indicates that the K_{IC} is highest when cracks propagate parallel to the poling direction and lowest when cracks grow perpendicular to the poling direction. The K_{IC} value of unpoled PZT falls between the two poled cases. Since local tensile and shear forces drive crack propagation, crack orientation is expected to be parallel to and 45° to the compression axis. For the poled PZT this means that crack growth occurs perpendicular to and at 45° to the poling direction (since the poling direction is perpendicular to the compression axis). Thus, the poled PZT is expected to have a K_{IC} value smaller than the

unpoled PZT. Assuming either that the PZTs have similar native flaw sizes, or that the native flaw population does not dominate failure, a smaller fracture toughness value equates to a lower strength.

All five of the materials have strengths that exceed the expected stress levels in naval transducers in use. For most devices the static compressive stress applied to the ceramic elements during fabrication does not exceed 82 MPa (12 ksi), although in a few designs the ceramic elements may be subjected to stresses as high as 345 MPa (50 ksi). The static stress is sufficiently large in magnitude to avoid the development of tensile stresses during excitation with an oscillating signal. Thus, the largest oscillating signal would cause stresses to vary from zero to twice the static compressive stress, or 164 to 690 MPa (24 to 100 ksi), depending upon transducer type. While the compressive strength of all five materials exceeded 690 MPa (100 ksi), the wide range of strengths in the PMNw/Ba, 877 ± 177 MPa (127 ± 26 ksi), is of concern.

Considerable differences are also seen in the stress-strain behavior of the five ceramics. The stress-strain curves of the two PZTs reveal nonlinear behavior (figure 3), which is more pronounced for the unpoled PZT. In piezoelectric materials mechanical loads can cause domains oriented parallel or nearly parallel to the compression axis to switch by 90°, which creates nonlinearity. Less switching is expected in the poled PZT compared to the unpoled PZT because the majority of the domains are already oriented perpendicular to the compression axis due to the poling process. In the unpoled PZT more 90° domain movement translates into a more pronounced nonlinear behavior, as seen in figure 3. The hysteresis in the stress-strain curves of the PZTs (figures 4 and 5) is also due to domain switching. When the mechanical stress is released some, but not all, of the domains switch back. The domains that do not switch back cause the remnant strain.

Compared to the PZTs, the nonlinear behavior of the unpoled PMN is quite small. At this time it is not understood why this is true. The electrostrictive PMNs are essentially linear to failure due to their lack of traditional domain structure.

All five materials were sensitive to changes in the strain rate. Research on alumina (reference 15) has linked this phenomenon to microplasticity. Intragranular twinning and slip were found to be responsible for initiating microcracks. Twinning and slip are strain-rate sensitive and diminish with increasing strain rate, thus leading to the strengthening. The strain associated with microplasticity is sufficiently small not to be reflected in the macroscopic stress-strain curve.

Decreasing amounts of microplasticity and consequently a smaller induced flaw population with increasing strain rate provide a feasible explanation for the strain rate sensitivity of the electrostrictive PMNs and the nearly linear unpoled PMN. The PZTs exhibit macroscopic nonlinear behavior due to domain motion; however, microplasticity cannot be ruled out as an explanation for PZTs as well. For both the unpoled and the poled PZT, the stress-strain curves from the three loading rates overlay within a region of scatter. Thus, changes in strain rate do not appear to have a noticeable affect on domain motion. Nonetheless, if microplastic processes, which are not detected in the macroscopic stress-strain curve, can lead to visible strain-rate

sensitivity, then subtle changes in domain motion may have a similar effect. However, it must be noted that the differences in the K_{IC} values of PZTs with different electrical histories are attributed to differences in the quantity of domain motion (references 11 through 14). Thus, if less domain motion means a smaller induced flaw population (and higher strength), it also means a smaller K_{IC} value (and lower strength). These conflicting effects lend plausibility to the possibility of other strain rate sensitive phenomena, such as twinning and slip, contributing to the development of the induced flaw population. This analysis also argues for a fracture process dependent upon an induced flaw population rather than the native flaw population.

SUMMARY

The five electroactive ceramics studied had a spectrum of strengths. The electrostrictive PMNs were the weakest with average strengths in the 870 MPa (126 ksi) range. They were followed in order of increasing strength by PZT poled perpendicular to the loading axis, unpoled PZT, and piezoelectric PMN. There was a 48% increase in strength from the electrostrictive PMNs to the piezoelectric PMN. It is argued that the differences in strength between all five materials are due mainly to differences in the fracture toughness value(s).

In each of the five electroactive ceramics, the compressive strength is a function of load (strain) rate similar to behavior seen in structural ceramics. The specific relationship is one in which strength increases with increasing strain rate. One feasible explanation involves the role of strain-rate-sensitive microplastic processes, such as twinning and slip. Microplasticity can initiate microcracks. Increasing the strain rate decreases the amount of microplasticity, which decreases the induced flaw population and consequently increases the strength. Detailed microstructural studies are needed to confirm this possibility.

The PZTs showed significant nonlinearity due to 90° domain switching. Hysteresis and a remnant strain existed upon unloading. The existence of a remnant strain was also attributed to the permanent switching of some of the 90° domains.

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